BOILING POINT DETERMINATION

FOR

AUTOMATE YELLOW 96

TO : U.S. Chemical Safety &
Hazard Investigation Board
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INTRODUCTION

The boiling point analysis of **Automate Yellow 96** was further investigative work that came about as a follow up to thermal analysis studies conducted on the sample. Initial correspondence between Mr. David Heller from the U.S. Chemical Safety & Hazard Investigation Board (USCSHIB) in Washington, DC, and Mr. Richard Wedlich of Chilworth Technology, Inc. focused around a report review of an incident that took place in Paterson, NJ. The site of the incident was the Morton plant during the synthesis of a dye product – Automate “Yellow 96”.

For purposes of boiling point determination, Chilworth Technology tested the pure liquid sample from Morton International, Inc. This report provides: (1) relevant background information; (2) a description of the test method; and (3) a discussion of the test results.

BACKGROUND

The boiling point of a liquid is the temperature at which the vapor pressure of the liquid is equal to the prevailing atmospheric pressure. The boiling point of a liquid at standard atmospheric pressure – 760 mm Hg -- is referred to as the normal boiling point. The boiling point provides an indication of the relative strength of intermolecular attractive forces in a liquid. Boiling point data are often necessary in the design of chemical processes for purposes of specifying operating conditions and materials handling practices.

METHOD

To perform boiling point analysis on **Automate Yellow 96,** Chilworth Technology first performed a very crude, small-scale screening to determine, roughly, at what temperature 1mL of sample would boil. The screening showed that the small sample had a very high boiling point (close to the decomposition temperature). Therefore, to conduct the testing as safely as possible, the boiling determination was made under vacuum conditions.

The boiling point apparatus consists of a round bottom flask (sample) with a thermometer well (to measure sample temperature) at a 45° angle, as well as a ground glass joint where the flask attaches to the rest of the distillation equipment. From the flask, glassware allows for a thermometer directly above the sample (to measure reflux temperature) and the connection to the condenser and distillate run-off. The high-potency vacuum is isolated from the distillation equipment via a ballast vessel (large glass flask). The ballast vessel connects the vacuum to the glassware, as well as provides a port for a vacuum meter. The digital gauge facilitates confident, accurate determinations of the vacuum pressure of the system.

First the entire system was evacuated to the desired vacuum pressure. The first trial at 1-2mbar was a closed system and that pressure proved to be the potency of the vacuum pump. The second trial conducted at 10mbar was performed using a needle valve opened very slightly to allow some air to bleed into the system. Then the sample was heated via a heat gun and an electric hot plate (which also provided magnetic stirring for a uniform sample temperature). As the sample approaches its boiling point temperature, some of the sample is vaporized. The condenser has 10° C coolant running through it and, therefore, condensation forms at the pointed bottom tip. As the sample comes closer and closer to reaching its boiling point, the reflux begins to drop. Reflux also begins forming on the thermometer directly above the sample. At the point where the reflux is produces drops at a constant rate and the temperature is no longer rising, sample has reached its boiling point. The temperature of the sample and the reflux are recorded and the pressure of the system is confirmed.

RESULTS

The results of boiling point determination trials are shown in **Table 1**. The boiling point of **Automate Yellow 96** was taken at two different pressures. At 1 – 2 mbar the boiling point was 130° C at the reflux and 138° C at the sample thermometer. Similarly, at 10mbar the sample was boiling at 185° C reflux and 193° C sample.

Boiling Point Determination

Sample Information

Company name	:	U.S. Chemical Safety & Hazard Investigation Board
Test powder	:	Automate Yellow 96
Ref. No.	:	7132 -
Origin of the sample	:	Morton International Inc.
Comment	:	Viscous, dark orange/yellow liquid

Test information

Test purpose	:	To determine the boiling point of Automate Yellow 96.
Apparatus	:	Round bottom flask (2), condenser (2), calibrated thermometers (2), chiller, ballast tank, vacuum, vacuum meter, Dewar flask, cold trap, & hot plate with stirrer.
Date of test	:	5/10/00
Operator	:	B. Carey

TABLE 1

TEST TRIAL	CONDITION	REFLUX TEMPERATURE	SAMPLE TEMPERATURE
1	1 – 2 mbar .76 – 1.52 mm Hg	130° C	138° C
2	10 mbar 7.6 mm Hg	185° C	193° C

BOILING POINT CORRECTION

Using the Pressure – Temperature Nomograph (located in the APPENDIX) from the Aldrich Chemical Catalog, a quick conversion can be made from a reduced-pressure boiling point to the boiling point at atmospheric conditions. As shown in **Table 2,** the two trials performed on Automate Yellow 96 at reduced pressures (experimental values) are in close agreement when converted to atmospheric boiling point (expected values). In the first trial, the reflux temperature was 130° C at 1 – 2 mbar or .76 to 1.52mm Hg. When corrected for atmospheric conditions of 760mm Hg the boiling point becomes 310° to 330° C. In the second trial, the boiling point is found to be 185° C at 10mbar or 7.6mm Hg. This converts to a boiling point of 330° C at 760mm Hg.

TABLE 2

TEST TRIAL	CONDITION	REFLUX TEMPERATURE	BOILING POINT CORRECTED FOR 760mm Hg
1	1 – 2 mbar .76 – 1.52 mm Hg	130° C	310° C to 330° C
2	10 mbar 7.60 mm Hg	185° C	330° C

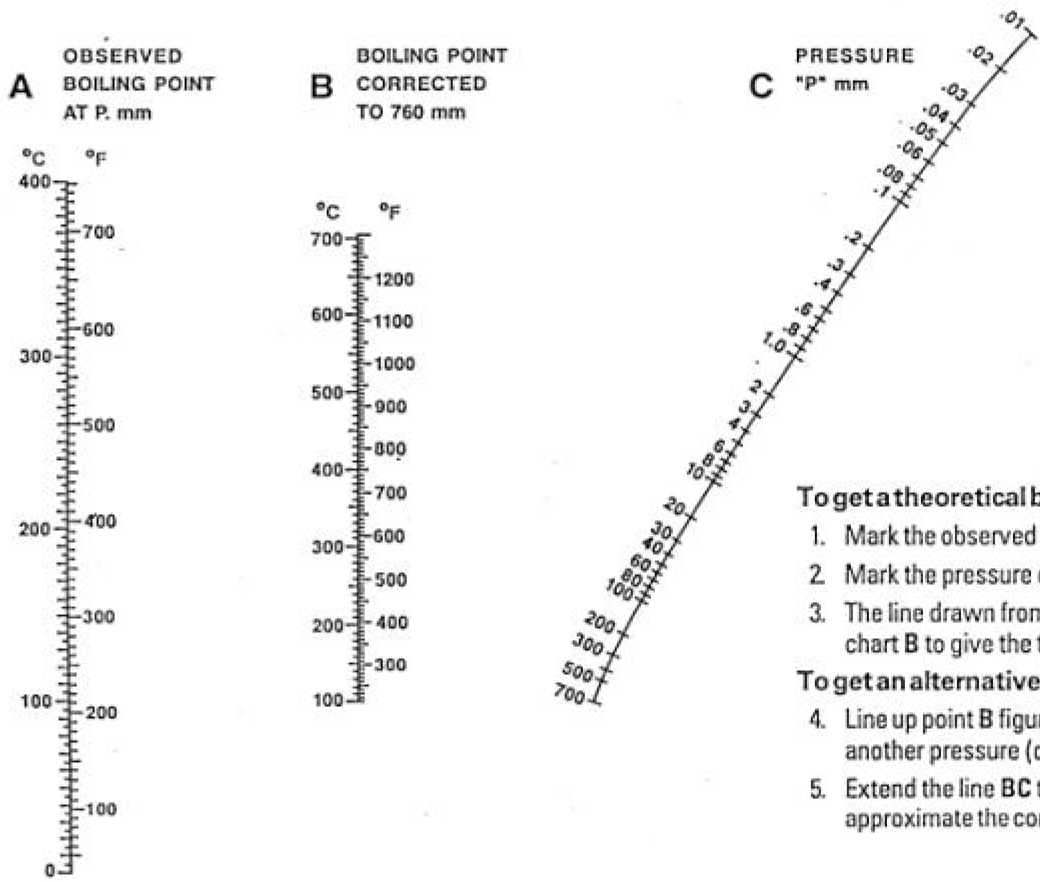
DISCUSSION

As was stated previously, an attempt was made to determine the boiling point of the sample at atmospheric conditions. From small-scale (1 mL) screening, it was observed that the boiling point of the sample was well within the range of the onset temperature for sample decomposition. Performing the analysis under vacuum conditions lowers the boiling point of the sample. The values determined by the boiling point analysis (performed under vacuum) can be plotted on a boiling point versus pressure graph and the value at atmospheric pressure can be extrapolated.

It is most likely that the sample will decompose before reaching the boiling point. Therefore, while boiling point data is useful in assessing the physical properties of **Automate Yellow 96,** one should place more attention on the thermal behavior analysis as the sample approaches its decomposition temperature.

A sample that is derived from multiple components is limited to the boiling point of its most volatile chemical. When determining a boiling point for a compound it is important to realize that one or more components of the sample may vaporize leaving, perhaps, a chemical with a very high boiling point and unique thermal properties of its own.

PRESSURE-TEMPERATURE NOMOGRAPH



To get a theoretical bp @ 760mm:

1. Mark the observed boiling point on chart **A**
2. Mark the pressure on chart **C**
3. The line drawn from point **A** to **C** intersects chart **B** to give the theoretical bp at 760mm

To get an alternative bp / pressure:

4. Line up point **B** figured in step 3 with another pressure (chart **C**)
5. Extend the line **BC** through chart **A** to approximate the corresponding bp